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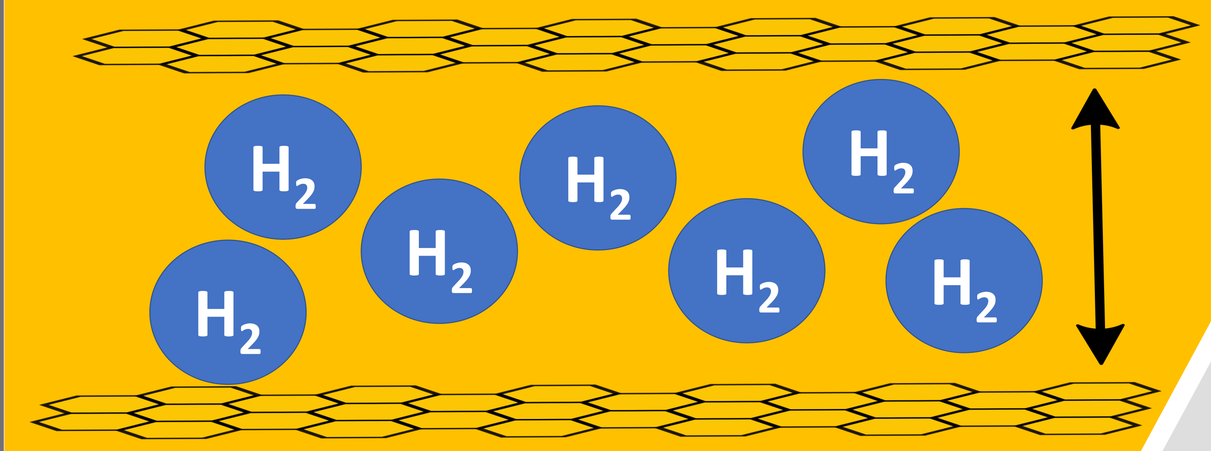
The State and Phase of Nanoconfined H_2

Neutron diffraction and spectroscopy study of H_2 adsorbed in micro and mesoporous activated carbons

Dr. LUI TERRY
VALESKA TING*
MI TIAN
SIMON BENDING
IVAN DA SILVA



Summary

Crystalline H_2 observed in activated carbons. FCC phase observed at elevated temperatures compared to bulk and in higher ratio in microporous carbon

Conclusion

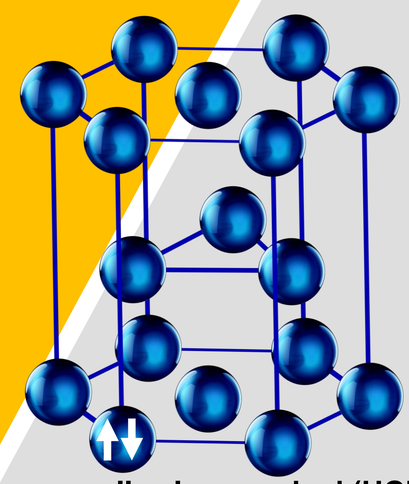
Nanoconfinement increases thermodynamic stability of FCC solid phase due to carbon – ortho H_2 interaction

Background

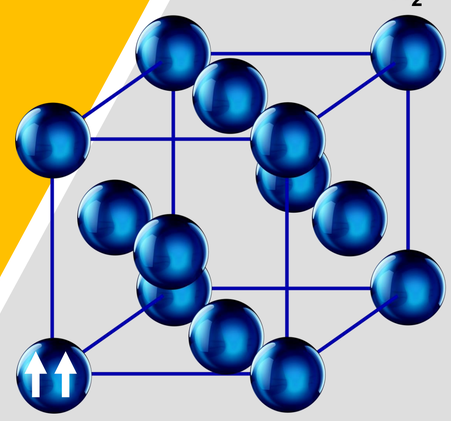
At low pressures and temperatures, two crystalline phases of H_2 are known: hexagonally close packed (HCP) and face-centred-cubic (FCC) H_2. FCC forms only when ortho concentration is >60 %. Recently it has been demonstrated that H_2 adsorbed into highly microporous carbon (< 2nm) becomes immobile and reaches densities above that of bulk liquid and solid above the critical temperature [1,2,3].
If this is true, then understanding the mechanism as to why and how these dense phases form would be vital to enhancing the storage capacity of these materials.

Aim

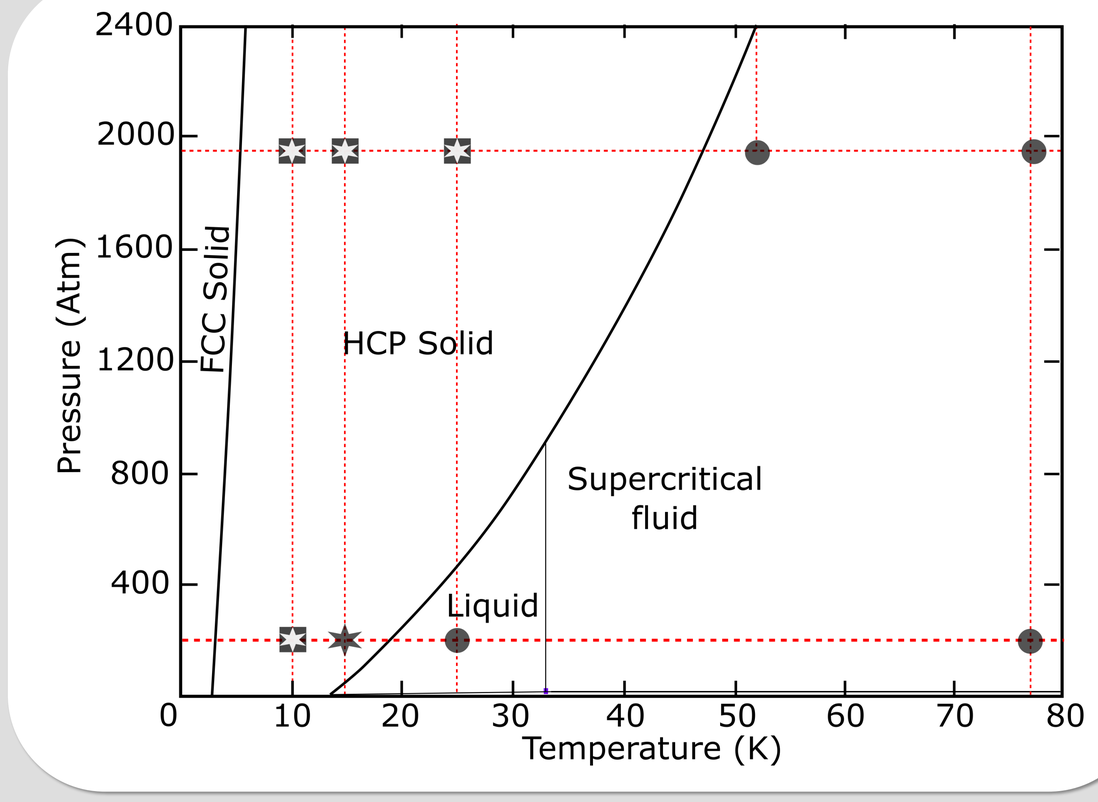
Determine the state & phase of H_2 confined in the pores of mesoporous (2-50 nm) Vs. microporous (< 2nm) activated carbons at temperatures across the bulk solid & liquid phase boundaries



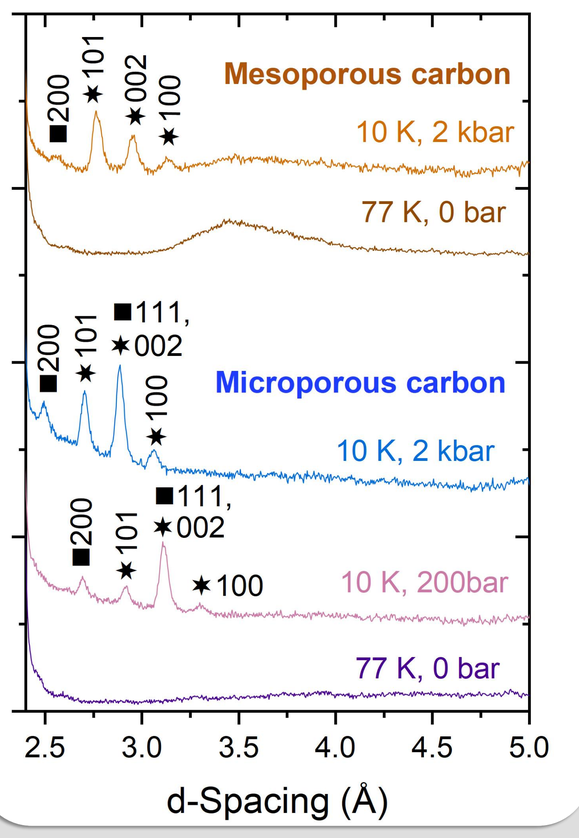
Hexagonally close packed (HCP) H_2 <60% ortho H_2



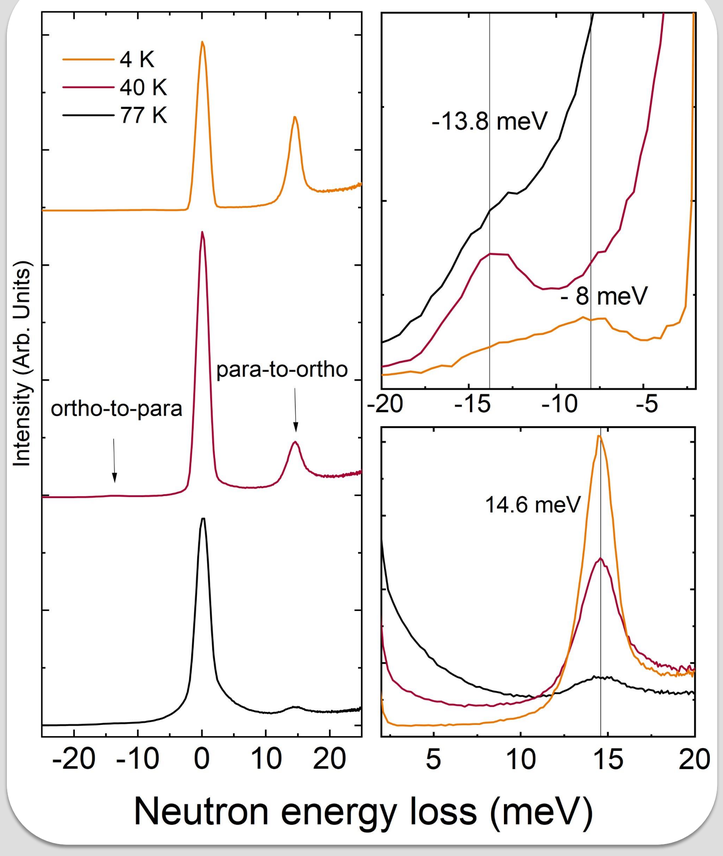
Face Centred Cubic (FCC) H_2 only forms when ortho-H_2 is >60%



Confined FCC H_2 observed at temperatures above the bulk: Bulk phase diagram of pure ortho H_2 overlaid with microporous carbon + H_2 neutron diffraction results. Red lines represent the experimental pressures and temperatures, squares represent FCC phase, stars represent HCP phase and filled circles short-range-order.



Increased FCC H_2 observed in microporous carbon: Neutron diffraction of microporous TE7 and mesoporous OLC porous carbons shows increased fraction of FCC phase (squares) over HCP phase (Stars) in microporous carbon.



Carbon - ortho-H_2 interaction: Temperature-dependent inelastic neutron scattering spectra of microporous carbon shows ortho-H_2 is rotationally hindered, whereas para-H_2 freely rotates, meaning ortho-H_2 is more strongly bound to the carbon.

Results

- Phase I HCP and FCC H_2 observed in both meso and microporous carbon.
- Microporous: up to 4.7 times more FCC to HCP
- Mesoporous: up to 0.3 times less FCC to HCP
- Confined FCC phase observed above bulk transition temperature
- Confined HCP obeys bulk transitions
- Confined H_2 unit cells are larger than bulk H_2
- Short-range-order observed in the bulk fluid range
- **Inelastic neutron scattering reveals confined ortho-H_2 is more strongly bound in carbon compared to para-H_2**

Methods and Materials

- Neutron Diffraction – GEM, ISIS - UK
- Inelastic Neutron Scattering - IN4, ILL - France
- Mesoporous OLC (modal pore width 9.9 nm)
- Microporous TE7 (modal pore width 0.55 nm)
- Degas, 350 C, 1x10^{-6} mbar, +8 hrs
- 99.999% n H_2

[1] Ting et al, ACS Nano, **9** (2015)
[2] Gallego et al, JACS, **133** (2011)
[3] Olsen et al, ACS Nano, **11** (2017)

University of BRISTOL
ACCIS ADVANCED COMPOSITES COLLABORATION FOR INNOVATION & SCIENCE
UNIVERSITY OF BATH
DEVCOM
LUI.TERRY@BRISTOL.AC.UK